

PATENT COOPERATION TREATY

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NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

United States Patent and Trademark
Office
(Box PCT)
Crystal Plaza 2
Washington, DC 20231
ÉTATS-UNIS D'AMÉRIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 21 July 1999 (21.07.99)	
International application No. PCT/US98/21287	Applicant's or agent's file reference 6010-6522
International filing date (day/month/year) 08 October 1998 (08.10.98)	Priority date (day/month/year) 09 October 1997 (09.10.97)
Applicant JONES, Gregory, M. et al	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:

23 April 1999 (23.04.99)

☐ in a notice effecting later election filed with the International Bureau on:2. The election ☒ was☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

<p>The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland</p> <p>Facsimile No.: (41-22) 740.14.35</p>	<p>Authorized officer</p> <p>Aino Metcalfe</p> <p>Telephone No.: (41-22) 338.83.38</p>
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(51) International Patent Classification ⁶ : G01N 29/02	A1	(11) International Publication Number: WO 99/19723
		(43) International Publication Date: 22 April 1999 (22.04.99)

(74) **Agents:** DUNLAP, Charles, E. et al.; Howell & Haferkamp, L.C., Suite 1400, 7733 Forsyth Boulevard, St. Louis, MO 63105 (US).

(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, HR, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

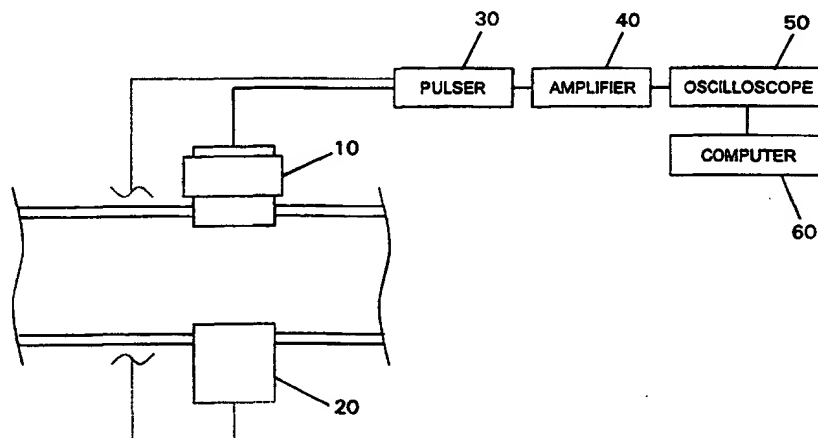
(63) Related by Continuation (CON) or Continuation-in-Part (CIP) to Earlier Application
 US 08/947,821 (CIP)
 Filed on 9 October 1997 (09.10.97)

(71) Applicant (for all designated States except US): BAKER
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(75) **Inventors/Applicants** (*for US only*): JONES, Gregory, M. [GB/GB]; 28 Bushbys Lane, Formby, Merseyside L37 2DZ (GB). POVEY, Malcolm, J., W. [GB/GB]; Greystones Farm, Bradshaw Lane, Huddersfield HD7 5UZ (GB). CAMPBELL, John [GB/GB]; 36 Cromptons Lane, Liverpool L18 3EX (GB).

Published
With international search report.

(54) **Title:** MEASUREMENT AND CONTROL OF ASPHALTENE AGGLOMERATION IN HYDROCARBON LIQUIDS



A method is provided for measuring the agglomerative state of asphaltenes in oil by applying an acoustic signal to the oil, detecting the scattered acoustic energy and using this detected signal to determine the relative particle size distribution of the asphaltene particles in the oil and/or their state of agglomeration. A method for controlling the agglomerative state of the asphaltenes which is based on the acoustic measurement technique is also provided.

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13. A method as set forth in claim 12, wherein the signal input probe and the sensor are located so that the signal direction of the probe intersects the signal direction of the sensor at an angle of less than about 60°.

14. A method as set forth in claim 13, wherein the signal input probe and the sensor are located so that the signal direction of the probe intersects the signal direction of the sensor at an angle of less than about 45°.

15. A method as set forth in claim 1, wherein the signal of acoustic energy is applied as a pulse and the step of resolving the magnitude of the detected scattered acoustic energy at selected frequencies within the selected frequency range comprises gating the detected scattered acoustic energy to that part of the detected energy
5 emanating from a focal region and Fourier transforming the detected scattered energy into a magnitude vs. frequency format.

16. A method as set forth in claim 1, wherein the signal of acoustic energy is applied as a tone-burst and the step of resolving the magnitude of the detected scattered acoustic energy at selected frequencies within the selected frequency range comprises detecting the magnitude of the scattered energy at selected frequencies
5 within the selected frequency range.

17. A method as set forth in claim 1, wherein determining the agglomerative state of the asphaltenes is effected by comparing the distribution of the asphaltene particles scattering acoustic energy within the selected frequency range with a standard.

18. A method as set forth in claim 17, wherein the standard is a sample of known particle size.

19. A method as set forth in claim 17, wherein the standard is a model of particle size based on scattering theory.

20. A method as set forth in claim 1, wherein the oil containing asphaltenes is in a process flow stream and the signal of acoustic energy is applied to the oil in the process flow stream.

21. A method for measuring the agglomerative state of asphaltenes in an oil containing asphaltenes comprising:

a. removing a sample of the oil and without diluting the oil;

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US98/21287

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) : G01N 29/02

US CL : 73/61.75

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 73/61.75, 64.41, 64.42, 61.71, 64.53, 53.05, 602, 599, 610, 611, 629, 865.5

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

APS

search terms: asphaltene, agglomeration, petroleum, particle

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5,132,225 A (DICKAKIAN) 21 July 1992 (21.07.92) see the abstract.	1-25
Y	De Boer et al, Screening of Crude Oils for Asphalt Precipitation: Theory, Practice, and the Selection of Inhibitors, February 1995, SPE Production & Facilities, pages 55-61.	1-25
Y	US 4,509,360 A (ERWIN et al) 09 April 1985 (09.04.85) col. 2 lines 58+	1-25
A	US 4,706,509 A (RIEBEL) 17 November 1997 (17.11.97) See entire document.	1-251
A	US 5,546,792 A (BECKER) 20 August 1996 (20.08.96) see entire document.	1-251

☒ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:	*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
A document defining the general state of the art which is not considered to be of particular relevance	*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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O document referring to an oral disclosure, use, exhibition or other means	
P document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search

10 DECEMBER 1998

Date of mailing of the international search report

14 JAN 1999

Name and mailing address of the ISA/US
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Authorized officer

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/US98/21287

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5,420,040 A (ANFINDSEN et al) 30 May 1995 (30.05.95) see entire document.	1-251

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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

REC'D 02 FEB 2000

WIPO PCT

Applicant's or agent's file reference 194-10244-PCT	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
International application No. PCT/US98/21287	International filing date (day/month/year) 08 OCTOBER 1998	Priority date (day/month/year) 09 OCTOBER 1997
International Patent Classification (IPC) or national classification and IPC IPC(6): and US Cl.: G01N 29/02 and 073/061.750		
Applicant BAKER HUGHES INCORPORATED		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 4 sheets.
- ☒ This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority. (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).
- These annexes consist of a total of 1 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of report with regard to novelty, inventive step or industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand 21 JULY 1999	Date of completion of this report 03 JANUARY 2000
Name and mailing address of the IPEA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230	Authorized officer HEZRON E. WILLY Telephone No. (703) 305-4705

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/US98/21287

I. Basis of the report

1. This report has been drawn on the basis of *(Substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments)*:

- ☐ the international application as originally filed.
- ☒ the description, pages (See Attached) , as originally filed.
pages _____ , filed with the demand.
pages _____ , filed with the letter of _____ .
pages _____ , filed with the letter of _____ .
- ☒ the claims, Nos. (See Attached) , as originally filed.
Nos. _____ , as amended under Article 19.
Nos. _____ , filed with the demand.
Nos. _____ , filed with the letter of _____ .
Nos. _____ , filed with the letter of _____ .
- ☒ the drawings, sheets/~~fig~~ (See Attached) , as originally filed.
sheets/~~fig~~ _____ , filed with the demand.
sheets/~~fig~~ _____ , filed with the letter of _____ .
sheets/~~fig~~ _____ , filed with the letter of _____ .

2. The amendments have resulted in the cancellation of:

- ☒ the description, pages NONE .
- ☒ the claims, Nos. NONE .
- ☒ the drawings, sheets/~~fig~~ NONE .

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the ~~Supplemental Box~~ Additional observations below (Rule 70.2(c)).

4. Additional observations, if necessary:

NONE

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/US98/21287

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**1. STATEMENT**

Novelty (N)	Claims	<u>1 - 26</u>	YES
	Claims	<u>NONE</u>	NO
Inventive Step (IS)	Claims	<u>1 - 26</u>	YES
	Claims	<u>NONE</u>	NO
Industrial Applicability (IA)	Claims	<u>1 - 26</u>	YES
	Claims	<u>NONE</u>	NO

2. CITATIONS AND EXPLANATIONS

Claims 1-26 meet the criteria set out in PCT Article 33(2)-(4), because the prior art does not teach or fairly suggest a method for **measuring the agglomerative state of asphaltenes in an oil sample known to contain asphaltenes** which radiates the oil sample with acoustic energy, and then detects the scattered acoustic energy from the oil sample over a selected frequency range so as to **resolve the magnitude of the detected scattered acoustic energy at selected frequencies within such selected frequency range**; which method is deemed by Examiner to not be suggested, taught or disclosed through any of the combination of US Patent No. 5132225 [by Dickakian], 4509360 [by Erwin et al.] and/or "Screening of Crude Oils for Asphalt Precipitation" [article by de Boer et al.] because as stated in Applicant's Response to Written Opinion of October 27, 1999 these references do not contain the application's instant step of resolving the magnitude of scattered acoustic energy at selected frequencies within a selected frequency range {i.e.- there is no **transformation of the back-scattered acoustic signal from an amplitude versus time signal into a magnitude versus frequency signal**}; nor do the same references contain therein a valid **motivational premise for combining the chromatography method of Dickakian with that acoustic method** of Erwin et al. and/or de Boer et al. as a mental guide in order to arrive at the instant invention- hence, the claimed subject matter per measurement and control of asphaltene agglomeration is deemed to contain novelty, to **constitute an inventive step {over the cited prior art or record}**, and to manifest a high degree of industrial applicability from the vantage point of one possessing ordinary skill in the art at the time of the invention, even for a skilled artisan or theoretician who was privy to wide ranges of all known analogous prior art during that time period.

----- NEW CITATIONS -----
none

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/US98/21287

Supplemental Box

(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: Boxes I - VIII

Sheet 10

I. BASIS OF REPORT:

This report has been drawn on the basis of the description,
pages. 1-23, as originally filed.

pages. NONE, filed with the demand.

and additional amendments:

not applicable

This report has been drawn on the basis of the claims,
numbers. 1-12 & 22-26, as originally filed.

numbers. NONE, as amended under Article 19.

numbers. NONE, filed with the demand.

and additional amendments:

Claims 13-21 filed with the letter of 01 November 1999

This report has been drawn on the basis of the drawings,
sheets. 1 - 10, as originally filed.

sheets. NONE, filed with the demand.

and additional amendments:

NONE